

(12)
United States Patent
Ling et al.

(10) **Patent No.:** **US 9,543,163 B2**
(45) **Date of Patent:** **Jan. 10, 2017**

(54)
METHODS FOR FORMING FEATURES IN A MATERIAL LAYER UTILIZING A COMBINATION OF A MAIN ETCHING AND A CYCLICAL ETCHING PROCESS

H01L 21/02164; H01L 21/02131; H01L 21/02063; H01L 21/31625; H01L 21/02126; H01L 21/0214; H01J 2237/334
(Continued)

(71)
Applicant: **Applied Materials, Inc.**, Santa Clara, CA (US)

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Inventors: **Mang-Mang Ling**, San Jose, CA (US); **Jungmin Ko**, Santa Clara, CA (US); **Sean S. Kang**, San Ramon, CA (US); **Jeremiah T. Pender**, San Jose, CA (US); **Srinivas D. Nemani**, Sunnyvale, CA (US); **Bradley Howard**, Pleasanton, CA (US)

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Primary Examiner — Nadine Norton
Assistant Examiner — Christopher Remavege
(74) *Attorney, Agent, or Firm* — Patterson & Sheridan, LLP

(73)
Assignee: **APPLIED MATERIALS, INC.**, Santa Clara, CA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21)
Appl. No.: **14/059,416**

(22)
Filed: **Oct. 21, 2013**

(65)
Prior Publication Data
US 2015/0056814 A1 Feb. 26, 2015

Related U.S. Application Data
(60)
Provisional application No. 61/868,037, filed on Aug. 20, 2013.

(51)
Int. Cl.
H01L 21/302 (2006.01)
H01L 21/311 (2006.01)
H01L 21/768 (2006.01)

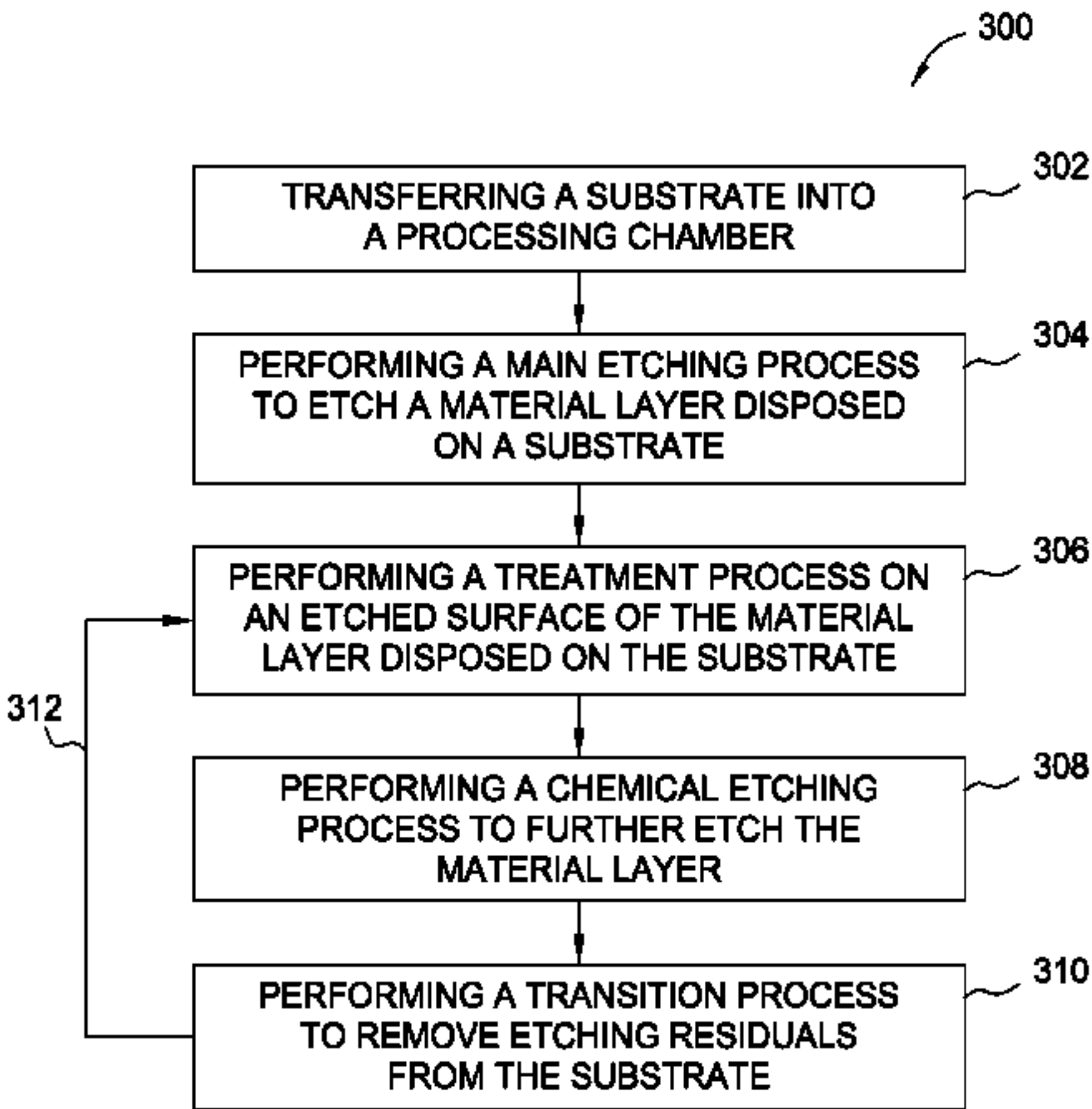
(52)
U.S. Cl.
CPC ... **H01L 21/31116** (2013.01); **H01L 21/76802** (2013.01); **H01J 2237/334** (2013.01)

(58)
Field of Classification Search
CPC H01L 21/02274; H01L 21/31116;

(57)
ABSTRACT

Methods for etching a material layer disposed on the substrate using a combination of a main etching step and a cyclical etching process are provided. The method includes performing a main etching process in a processing chamber to an oxide layer, forming a feature with a first predetermined depth in the oxide layer, performing a treatment process on the substrate by supplying a treatment gas mixture into the processing chamber to treat the etched feature in the oxide layer, performing a chemical etching process on the substrate by supplying a chemical etching gas mixture into the processing chamber, wherein the chemical etching gas includes at least an ammonium gas and a nitrogen trifluoride, wherein the chemical etching process further etches the feature to a second predetermined depth, and performing a transition process on the etched substrate by supplying a transition gas mixture into the processing chamber.

19 Claims, 4 Drawing Sheets



(58) **Field of Classification Search**
USPC 257/E21.029, E21.252, E21.577
See application file for complete search history.

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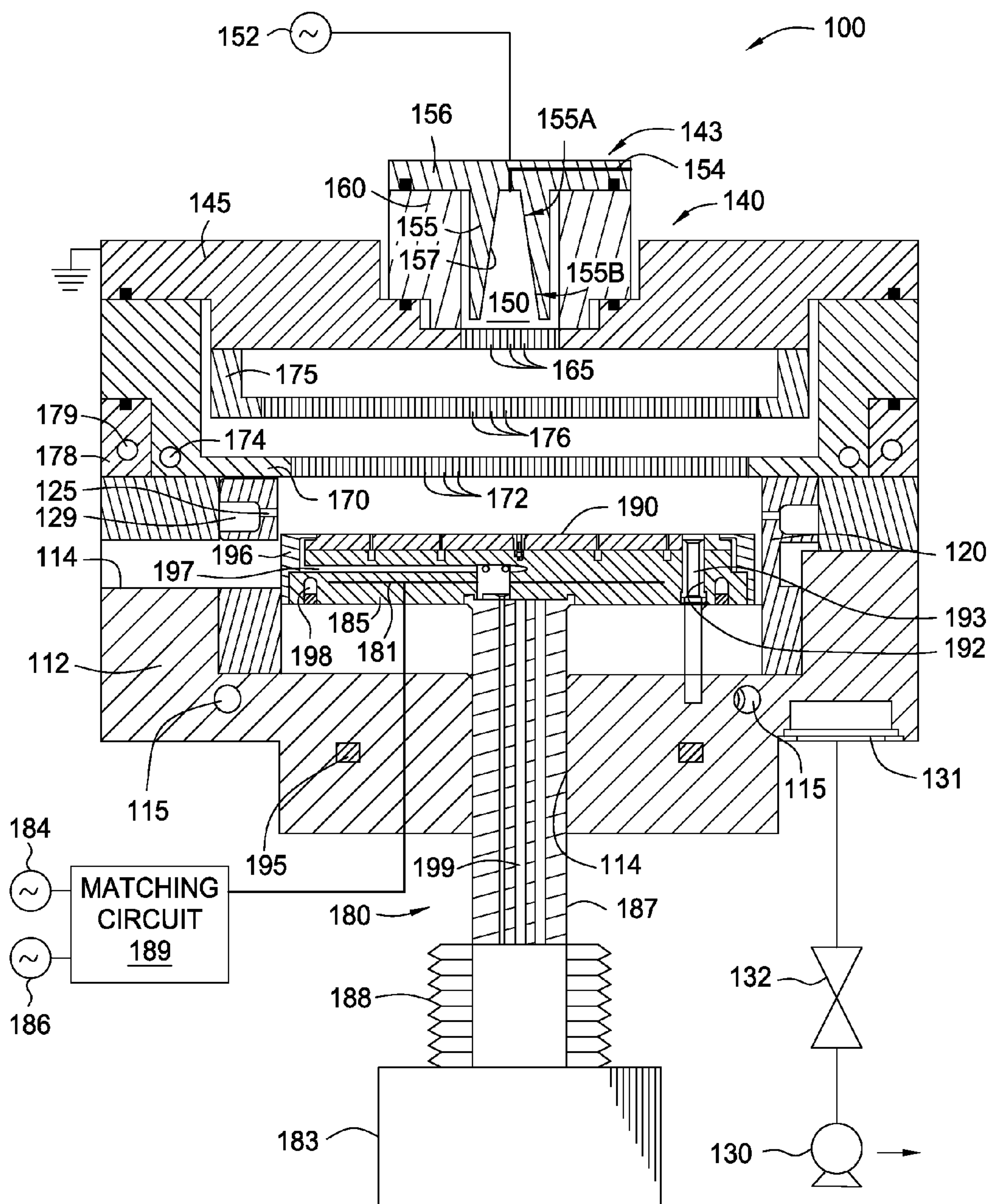


FIG. 1

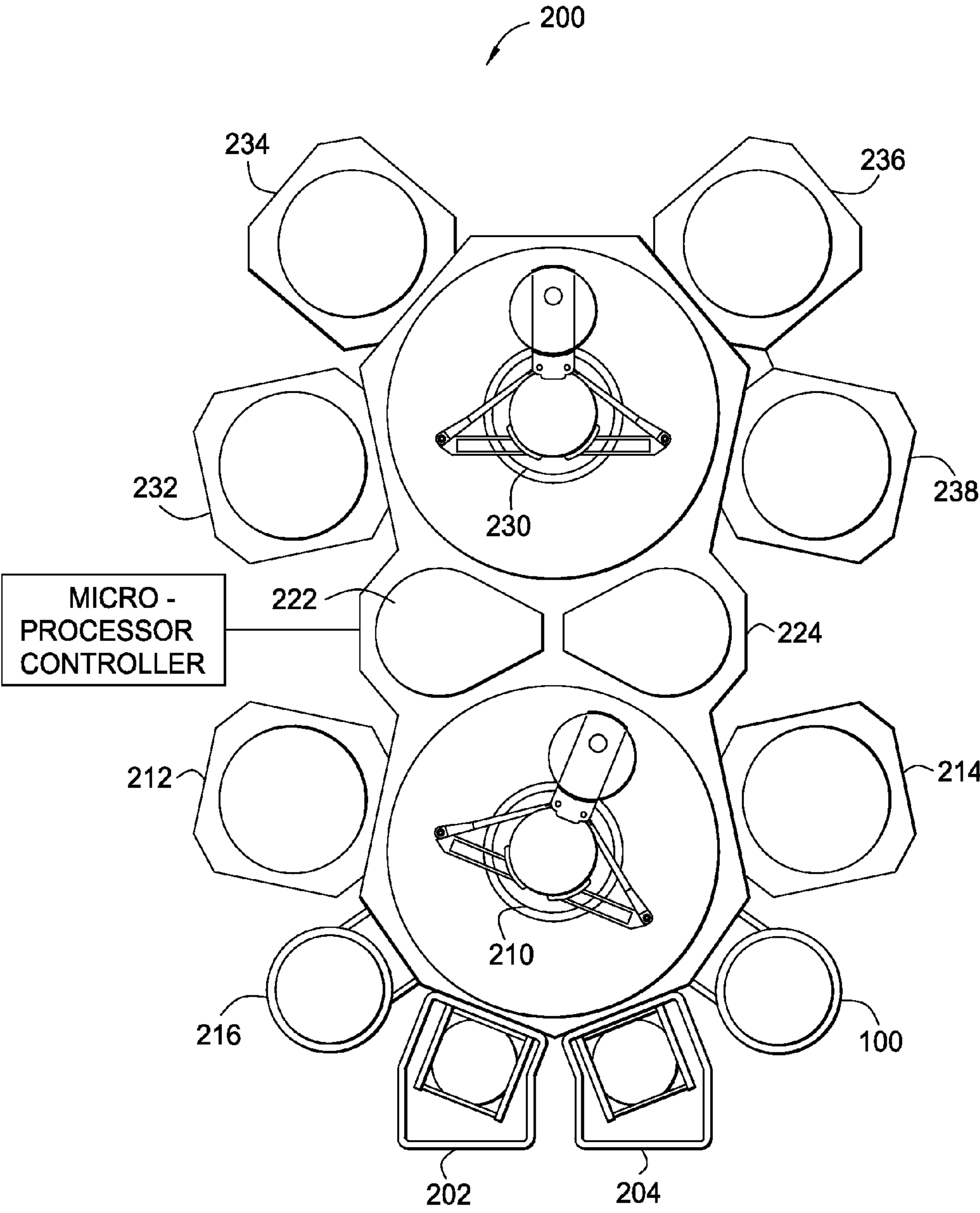


FIG. 2

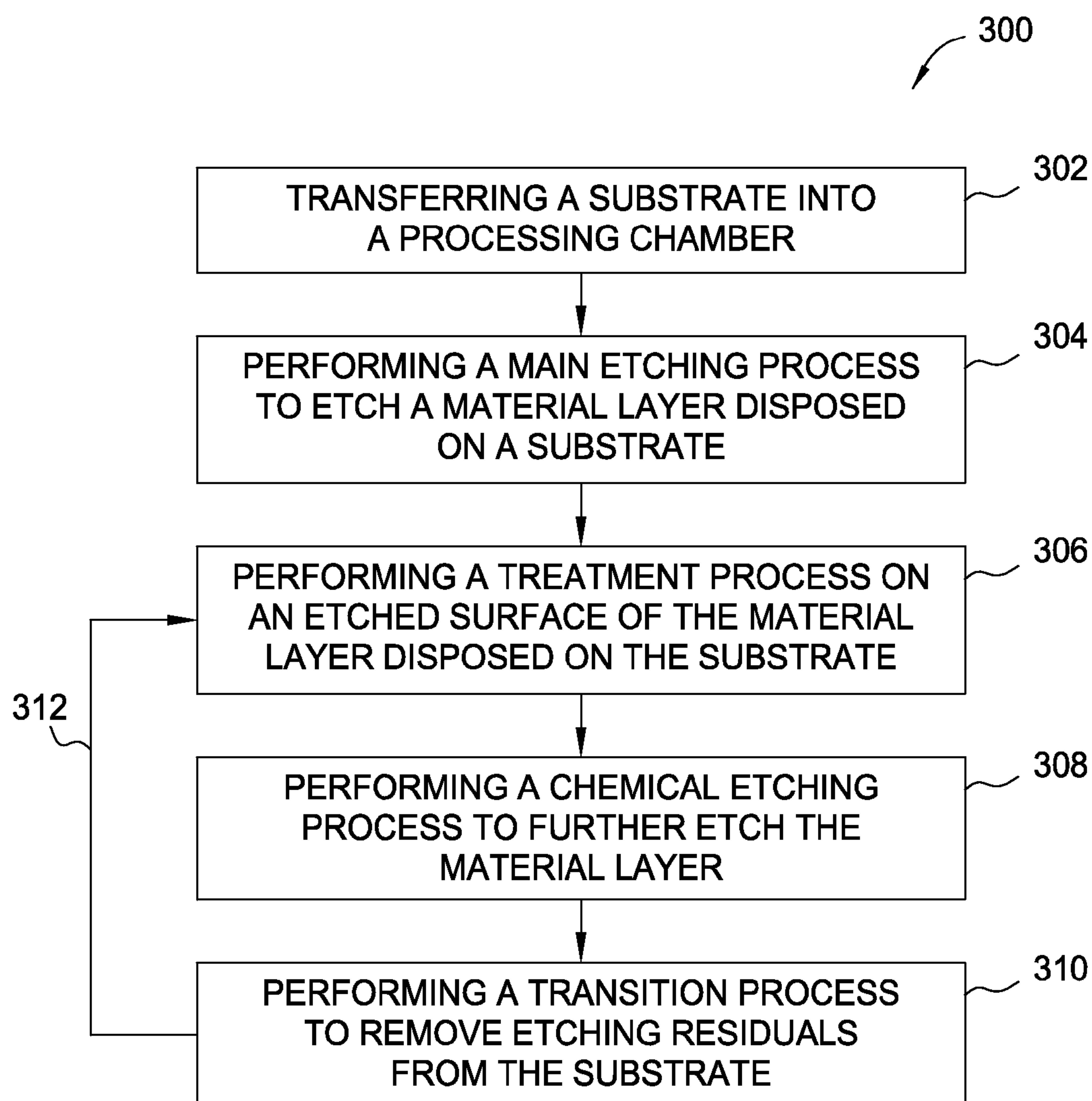


FIG. 3

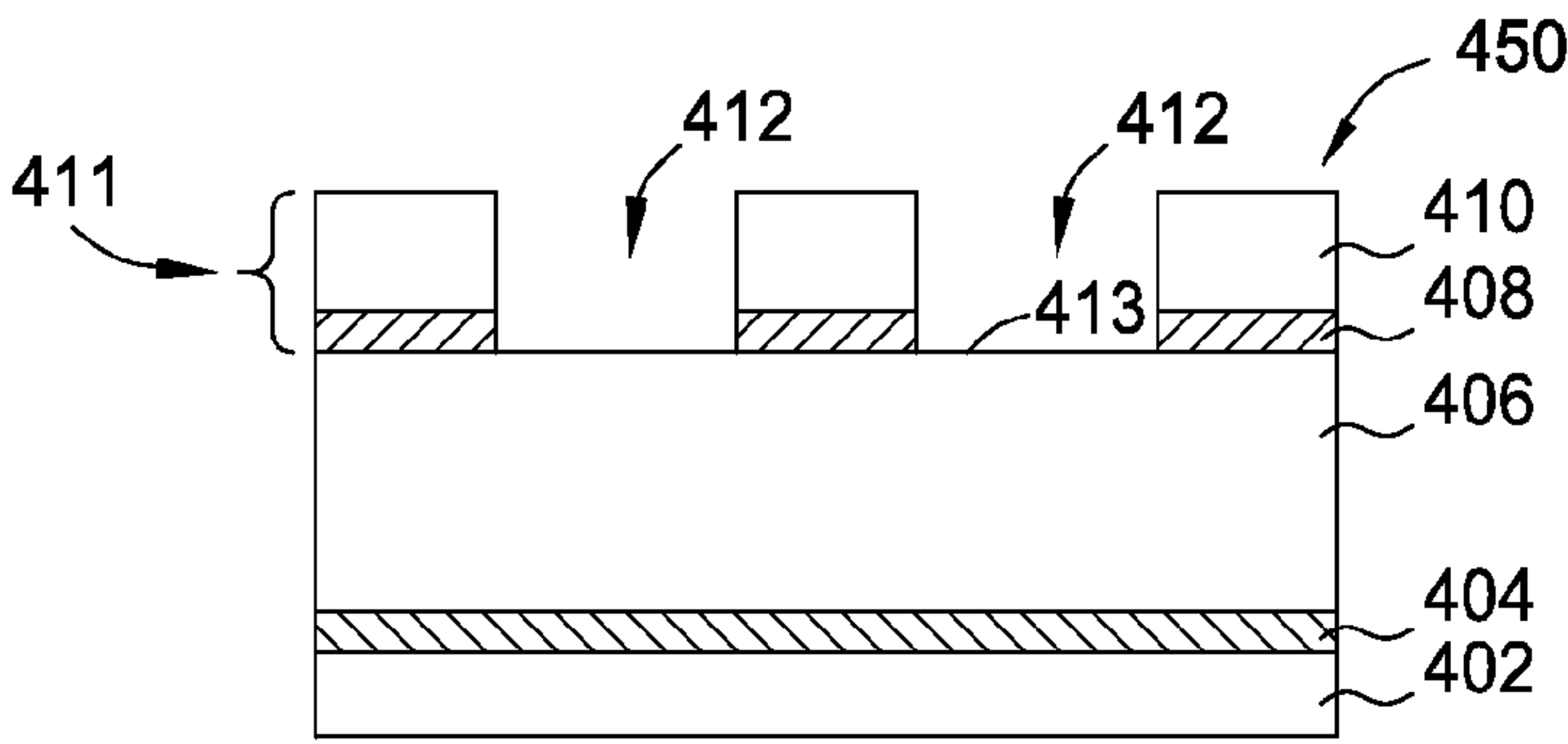


FIG. 4A

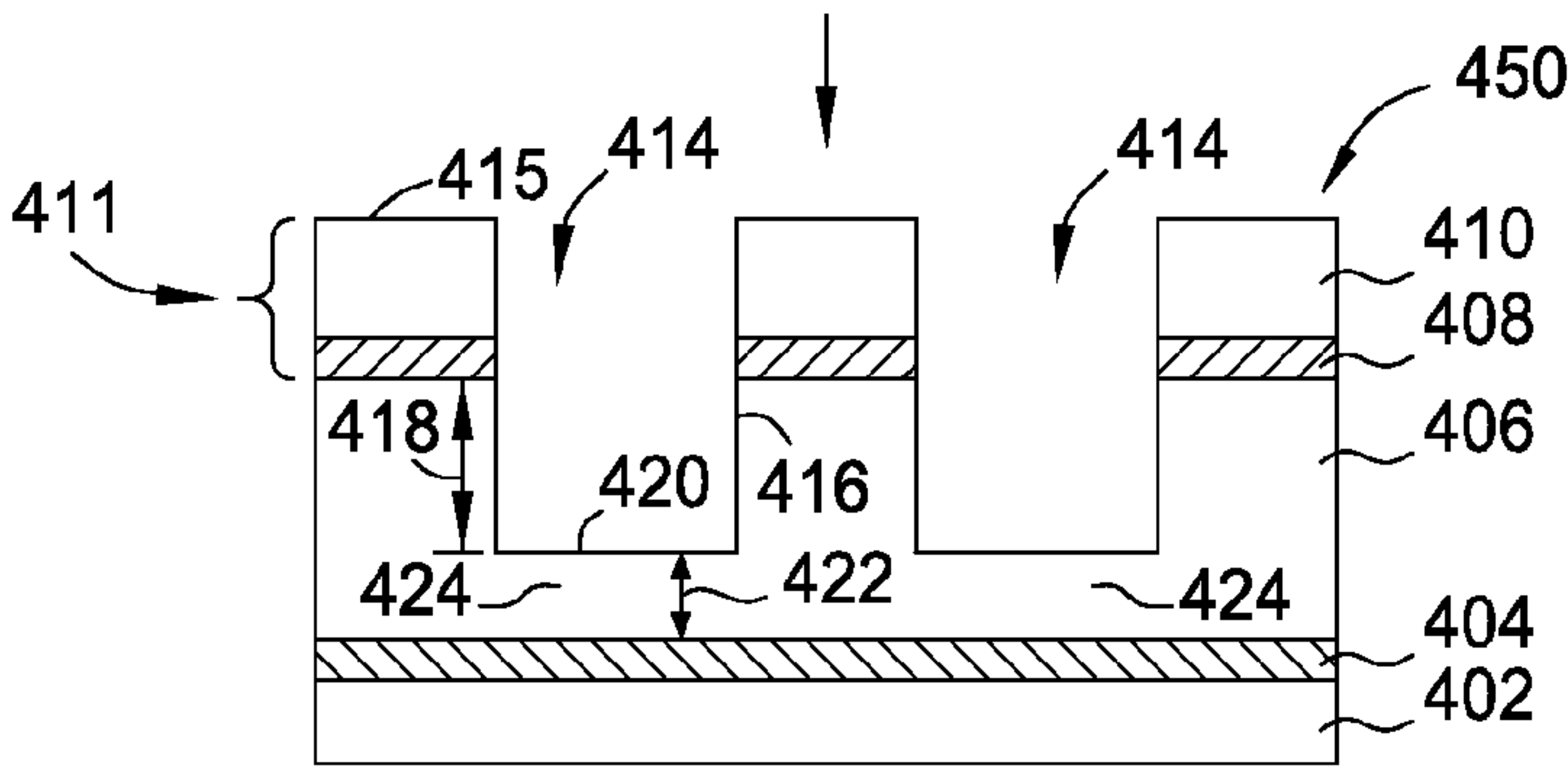


FIG. 4B

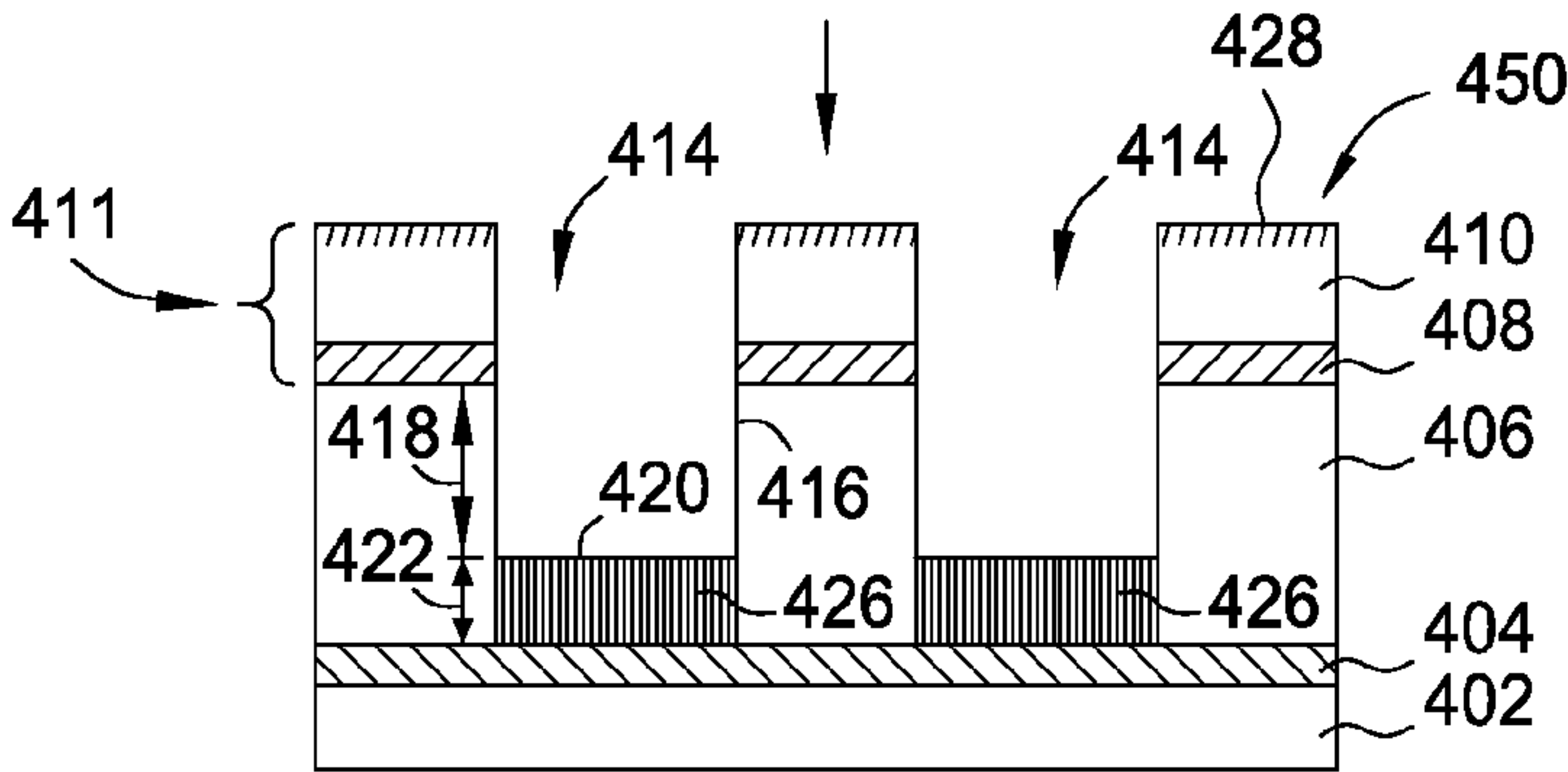


FIG. 4C

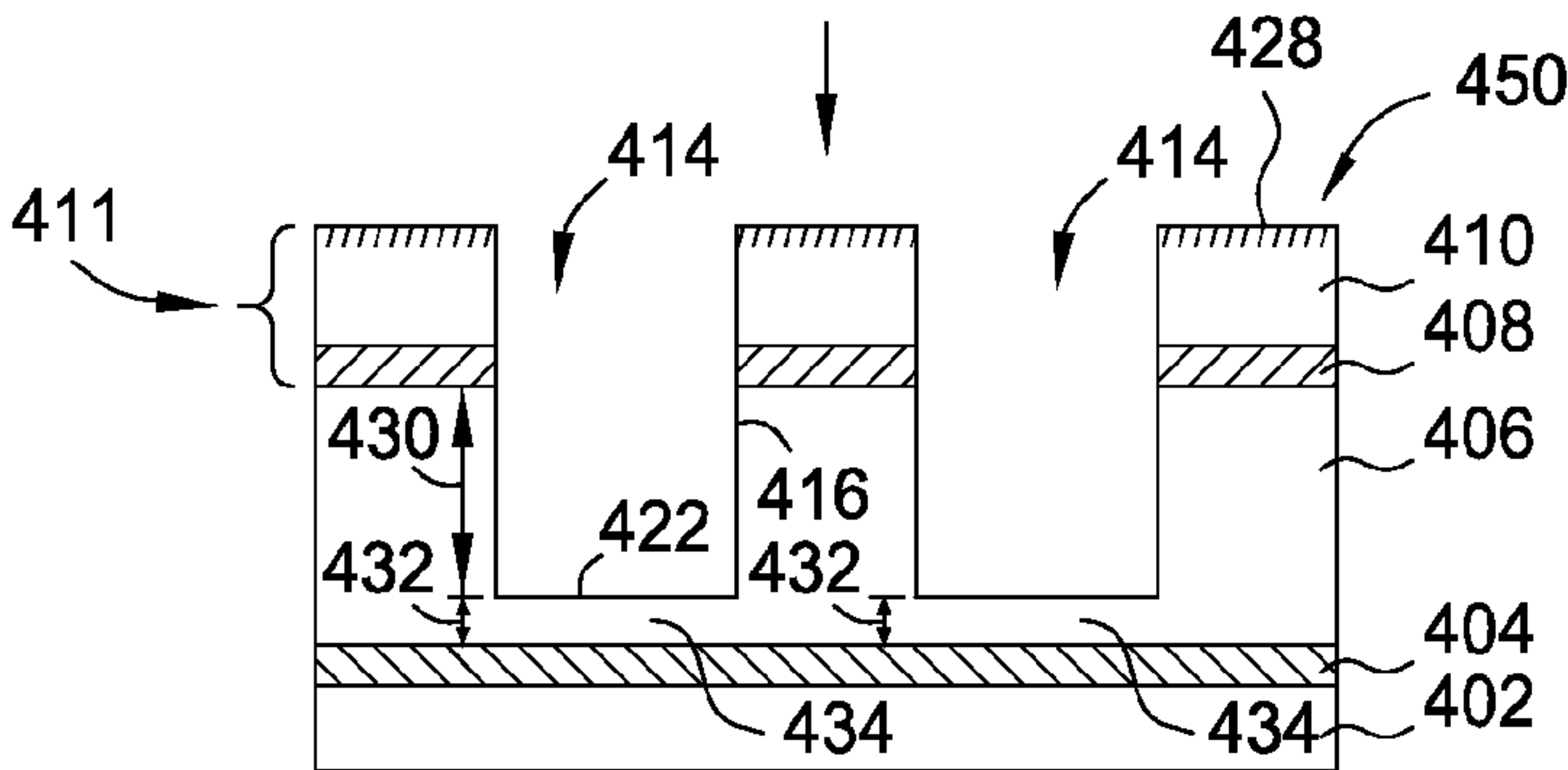


FIG. 4D

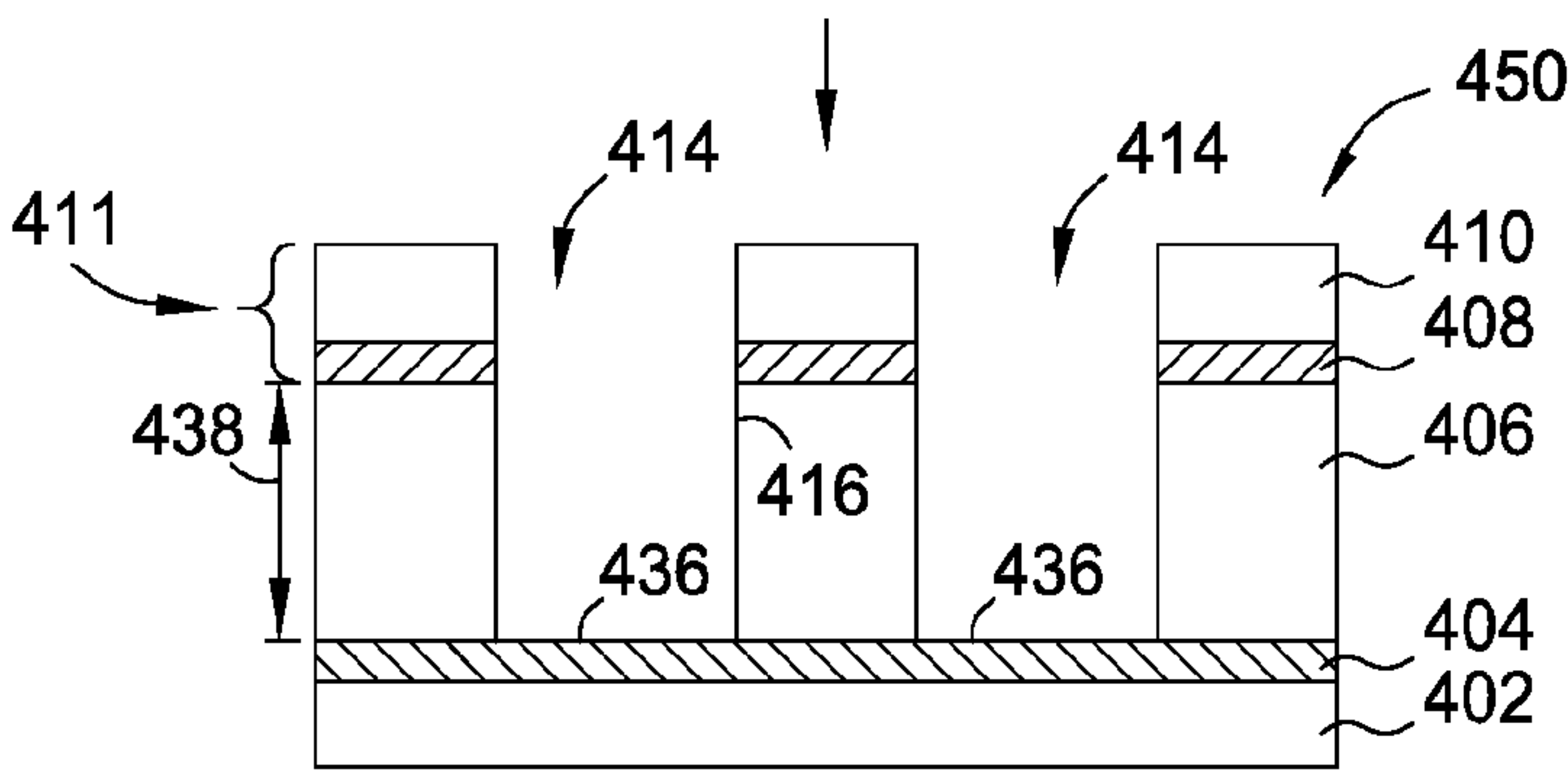


FIG. 4E

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METHODS FOR FORMING FEATURES IN A MATERIAL LAYER UTILIZING A COMBINATION OF A MAIN ETCHING AND A CYCLICAL ETCHING PROCESS

CROSS REFERENCE TO RELATED APPLICATIONS

This application claims benefit of U.S. Provisional Application Ser. No. 61/868,037, filed Aug. 20, 2013, which is incorporated by reference in its entirety.

BACKGROUND

Field

Embodiments of the present invention generally relate to methods for forming semiconductor devices. More particularly, embodiments of the present invention generally relate to methods for etching a material layer disposed on a substrate using a combination of a main etching and a cyclically etching process for manufacturing semiconductor devices.

Description of the Related Art

Reliably producing sub-half micron and smaller features is one of the key technology challenges for next generation very large scale integration (VLSI) and ultra large-scale integration (ULSI) of semiconductor devices. However, as the limits of circuit technology are pushed, the shrinking dimensions of VLSI and ULSI interconnect technology have placed additional demands on processing capabilities. Reliable formation of gate structures on the substrate is important to VLSI and ULSI success and to the continued effort to increase circuit density and quality of individual substrates and die.

A patterned mask, such as a photoresist layer, is commonly used in forming structures, such as gate structure, shallow trench isolation (STI), bit lines and the like, on a substrate by an etching process. The patterned mask is conventionally fabricated by using a lithographic process to optically transfer a pattern having the desired critical dimensions to a layer of photoresist. The photoresist layer is then developed to remove undesired portion of the photoresist, thereby creating openings in the remaining photoresist.

In order to enable fabrication of next generation devices and structures, the geometry limits of the structures designed to be formed for the semiconductor devices has been pushed against technology limits, the need for accurate process control for the manufacture of small critical dimensional structures with high aspect ratio has become increasingly important. Poor process control during etching process will result in irregular structure profiles and line edge roughness, thereby resulting in poor line integrity of the formed structures. Additionally, irregular profiles and growth of the etching by-products formed during etching may gradually block the small openings used to fabricate the small critical dimension structures, thereby resulting in bowed, distorted, toppled, or twisted profiles of the etched structures.

Furthermore, the similarity between the materials selected for the hardmask layer and the adjacent layers disposed in the film stack, and even the underlying material on the substrate, may also result in similar etch properties therebetween, thereby resulting in poor selectivity during etching. Poor selectivity between the hardmask layer, adjacent layers and the materials on the substrate may result in non-uniform, tapered and deformed profile of the hardmask layer, thereby leading to poor pattern transfer and failure of accurate structure dimension control. The chemical etchant used in

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the etch process is required to have a greater etch selectivity for the material layers in the film stack. That is, the chemical etchant etches the one or more layers of the film stack at a rate much faster than the energy sensitive resist or the materials disposed on the substrate. The etch selectivity to the one or more material layers of the film stack over the resist prevents the energy sensitive resist from being consumed prior to completion of the pattern transfer. Thus, a highly selective etchant enhances accurate pattern transfer. However, conventional etchants are not selective enough to enable robust manufacturing of next generation devices.

Thus, there is a need for improved methods for etching a material layer with high aspect ratio for manufacturing semiconductor devices with high selectivity and accurate process and profile control.

SUMMARY

Methods for etching a material layer disposed on the substrate using a combination of a main etching step and a cyclically etching process are provided. In one embodiment, a method for etching a material layer disposed on a substrate includes performing a main etching process in a processing chamber to etch an oxide layer exposed by a patterned mask layer disposed on a substrate, forming a feature with a first predetermined depth in the oxide layer, performing a treatment process on the substrate by supplying a treatment gas mixture into the processing chamber to treat the etched feature in the oxide layer, performing a chemical etching process on the substrate by supplying a chemical etching gas mixture into the processing chamber, wherein the chemical etching gas includes at least an ammonium gas and a nitrogen trifluoride, wherein the chemical etching process further etches the feature to a second predetermined depth, and performing a transition process on the etched substrate by supplying a transition gas mixture into the processing chamber.

In another embodiment, a method for etching a feature in a material layer includes transferring a substrate having a material layer disposed thereon into a processing chamber, wherein the material layer has a patterned mask layer disposed thereon exposing a portion of the material layer, supplying an etching gas mixture into the processing chamber to etch the portion of the material layer exposed by the patterned mask layer to form a feature in the material layer until to a first predetermined depth of the feature is formed in the material layer, wherein the etching gas mixture includes a halogenated carbon gas, supplying a treatment gas mixture to treat the feature formed in the material layer, wherein the treatment gas mixture includes an inert gas, and supplying a chemical etching gas mixture into the processing chamber, wherein the chemical etching gas mixture includes at least an ammonium gas and a nitrogen trifluoride, and supplying a transition gas mixture to the processing chamber, wherein the transition gas mixture includes an inert gas.

In yet another embodiment, a method for etching a feature in a material layer includes transferring a substrate having a material layer disposed thereon into a processing chamber, wherein the material layer has a patterned mask layer disposed thereon exposing a portion of the material layer, supplying a C_4F_8 or C_4F_6 gas into the processing chamber to etch the portion of the material layer exposed by the patterned mask layer to form a feature in the material layer until to a first predetermined depth of the feature is formed in the material layer, wherein the first predetermined depth of the feature is between about 15 percent and about 25 percent to

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a total thickness of the material layer, supplying a Ar or He gas while applying a first RF bias power to treat the feature formed in the material layer, and supplying a chemical etching gas mixture including at least an ammonium gas and a nitrogen trifluoride while applying a first RF source power to etch the treated feature, and supplying a Ar or He gas to the processing chamber without applying a RF power.

BRIEF DESCRIPTION OF THE DRAWINGS

So that the manner in which the above recited features of the present invention can be understood in detail, a more particular description of the invention, briefly summarized above, can be had by reference to embodiments, some of which are illustrated in the appended drawings. It is to be noted, however, that the appended drawings illustrate only typical embodiments of this invention and are therefore not to be considered limiting of its scope, for the invention can admit to other equally effective embodiments.

FIG. 1 is a cross section view of an illustrative processing chamber in which embodiments of the invention may be practiced.

FIG. 2 is a schematic top-view diagram of an illustrative multi-chamber processing system.

FIG. 3 depicts a flow diagram for etching a material layer using a cyclical etching process in accordance with one embodiment of the present invention; and

FIGS. 4A-4E depict cross-sectional views of a material layer disposed on a semiconductor substrate during the etching of the material layer in accordance with one embodiment of the present invention.

To facilitate understanding, identical reference numerals have been used, where possible, to designate identical elements that are common to the figures. It is contemplated that elements and features of one embodiment may be beneficially incorporated in other embodiments without further recitation.

It is to be noted, however, that the appended drawings illustrate only exemplary embodiments of this invention and are therefore not to be considered limiting of its scope, for the invention may admit to other equally effective embodiments.

DETAILED DESCRIPTION

Embodiments of the present invention provides methods for etching a material layer for high aspect ratio features formed on a substrate for semiconductor devices, with high selectivity and accurate profile control. In one embodiment, the etching process includes using a first step of a main etching process along with a second step of slow cyclical etching process to gradually etch the material layer until the underling substrate is exposed. The slow cyclical etching process may assist controlling the etching selectivity and providing a relatively accurate etch stop endpoint at the interface so as to provide a good profile control for the features formed in the material layer. In one embodiment, the etching process may be utilized to form features, trenches, or vias in a contact dielectric layer for a contact structure for semiconductor devices.

The term "contact structure" as used herein refers to a layer of material that includes a metal silicide that can form part of a gate electrode. In one or more embodiments, the metal silicide can be nickel silicide, cobalt silicide, titanium silicide or any combination thereof. The metal silicide can also include tungsten, tungsten silicide, Ti/Co alloy silicide, Ti/Ni alloy silicide, Co/Ni alloy silicide and Ni/Pt silicide.

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The contact structure may alternatively be silicon based, germanium based or silicon based with germanium dopant and/or other dopant.

FIG. 1 is a cross sectional view of an illustrative processing chamber 100 suitable for conducting an etching process as further described below. The chamber 100 may be configured to remove materials from a material layer disposed on a substrate surface. The chamber 100 is particularly useful for performing the plasma assisted dry etch process. The processing chamber 100 may be a Siconi™ chamber which is available from Applied Materials, Santa Clara, Calif. It is noted that other vacuum processing chambers available from other manufactures may also be utilized to practice the present invention.

The processing chamber 100 provides both heating and cooling of a substrate surface without breaking vacuum. In one embodiment, the processing chamber 100 includes a chamber body 112, a lid assembly 140, and a support assembly 180. The lid assembly 140 is disposed at an upper end of the chamber body 112, and the support assembly 180 is at least partially disposed within the chamber body 112.

The chamber body 112 includes a slit valve opening 114 formed in a sidewall thereof to provide access to the interior of the processing chamber 100. The slit valve opening 114 is selectively opened and closed to allow access to the interior of the chamber body 112 by a wafer handling robot (not shown).

In one or more embodiments, the chamber body 112 includes a channel 115 formed therein for flowing a heat transfer fluid therethrough. The heat transfer fluid can be a heating fluid or a coolant and is used to control the temperature of the chamber body 112 during processing and substrate transfer. Control of the temperature of the chamber body 112 is important to prevent unwanted condensation of the gas or byproducts on the chamber walls. Exemplary heat transfer fluids include water, ethylene glycol, or a mixture thereof. An exemplary heat transfer fluid may also include nitrogen gas.

The chamber body 112 can further include a liner 120 that surrounds the support assembly 180. The liner 120 is removable for servicing and cleaning. The liner 120 can be made of a metal such as aluminum, a ceramic material, or any other process compatible material. The liner 120 can be bead blasted to increase surface roughness and/or surface area which increases the adhesion of any material deposited thereon, thereby preventing flaking of material which results in contaminants of the processing chamber 100. In one or more embodiments, the liner 120 includes one or more apertures 125 and a pumping channel 129 formed therein that is in fluid communication with a vacuum system. The apertures 125 provide a flow path for gases into the pumping channel 129, which provides an egress for the gases within the processing chamber 100.

The vacuum system can include a vacuum pump 130 and a throttle valve 132 to regulate flow of gases through the processing chamber 100. The vacuum pump 130 is coupled to a vacuum port 131 disposed in the chamber body 112 and therefore, in fluid communication with the pumping channel 129 formed within the liner 120. The terms "gas" and "gases" are used interchangeably, unless otherwise noted, and refer to one or more precursors, reactants, catalysts, carrier, purge, cleaning, combinations thereof, as well as any other fluid introduced into the chamber body 112.

The lid assembly 140 includes at least two stacked components configured to form a plasma volume or cavity therebetween. In one or more embodiments, the lid assembly 140 includes a first electrode 143 ("upper electrode") dis-

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posed vertically above a second electrode **145** (“lower electrode”) confining a plasma volume or cavity **150** therebetween. The first electrode **143** is connected to a power source **152**, such as an RF power supply, and the second electrode **145** is connected to ground, forming a capacitance between the two electrodes **143,145**.

In one or more embodiments, the lid assembly **140** includes one or more gas inlets **154** (only one is shown) that are at least partially formed within an upper section **156** of the first electrode **143**. The one or more process gases enter the lid assembly **140** via the one or more gas inlets **154**. The one or more gas inlets **154** are in fluid communication with the plasma cavity **150** at a first end thereof and coupled to one or more upstream gas sources and/or other gas delivery components, such as gas mixers, at a second end thereof.

In one or more embodiments, the first electrode **143** has an expanding section **155** that houses the plasma cavity **150**. In one or more embodiments, the expanding section **155** is an annular member that has an inner surface or diameter **157** that gradually increases from an upper portion **155A** thereof to a lower portion **155B** thereof. As such, the distance between the first electrode **143** and the second electrode **145** is variable. That varying distance helps control the formation and stability of the plasma generated within the plasma cavity **150**.

In one or more embodiments, the expanding section **155** resembles an inverted truncated cone or “funnel.” In one or more embodiments, the inner surface **157** of the expanding section **155** gradually slopes from the upper portion **155A** to the lower portion **155B** of the expanding section **155**. The slope or angle of the inner diameter **157** can vary depending on process requirements and/or process limitations. The length or height of the expanding section **155** can also vary depending on specific process requirements and/or limitations.

As mentioned above, the expanding section **155** of the first electrode **143** varies the vertical distance between the first electrode **143** and the second electrode **145** because of the gradually increasing inner surface **157** of the first electrode **143**. That variable distance is directly related to the power level within the plasma cavity **150**. Not wishing to be bound by theory, the variation in distance between the two electrodes **143, 145** allows the plasma to find the necessary power level to sustain itself within some portion of the plasma cavity **150** if not throughout the entire plasma cavity **150**. The plasma within the plasma cavity **150** is therefore less dependent on pressure, allowing the plasma to be generated and sustained within a wider operating window. As such, a more repeatable and reliable plasma can be formed within the lid assembly **140**. As the plasma generated in the plasma cavity **150** is defined in the lid assembly **140** prior to entering into a processing region above the support assembly **180** wherein the substrate is processed, the plasma is considered as a remote plasma source that generated remotely from the processing region.

The expanding section **155** is in fluid communication with the gas inlet **154** as described above. The first end of the one or more gas inlets **154** can open into the plasma cavity **150** at the upper most point of the inner diameter of the expanding section **155**. Similarly, the first end of the one or more gas inlets **154** can open into the plasma cavity **150** at any height interval along the inner diameter **157** of the expanding section **155**. Although not shown, two gas inlets **154** can be disposed at opposite sides of the expanding section **155** to create a swirling flow pattern or “vortex” flow into the expanding section **155** which helps mix the gases within the plasma cavity **150**.

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The lid assembly **140** can further include an isolator ring **160** that electrically isolates the first electrode **143** from the second electrode **145**. The isolator ring **160** can be made from aluminum oxide or any other insulative, process compatible material. The isolator ring **160** surrounds or substantially surrounds at least the expanding section **155**.

The lid assembly **140** can further include a distribution plate **170** and blocker plate **175** adjacent the second electrode **145**. The second electrode **145**, distribution plate **170** and blocker plate **175** can be stacked and disposed on a lid rim **178** which is connected to the chamber body **112**. A hinge assembly (not shown) can be used to couple the lid rim **178** to the chamber body **112**. The lid rim **178** can include an embedded channel or passage **179** for housing a heat transfer medium. The heat transfer medium can be used for heating, cooling, or both, depending on the process requirements.

In one or more embodiments, the second electrode or top plate **145** can include a plurality of gas passages or apertures **165** formed beneath the plasma cavity **150** to allow gas from the plasma cavity **150** to flow therethrough. The distribution plate **170** is substantially disc-shaped and also includes a plurality of apertures **172** or passageways to distribute the flow of gases therethrough. The apertures **172** can be sized and positioned about the distribution plate **170** to provide a controlled and even flow distribution to the chamber body **112** where the substrate to be processed is located. Furthermore, the apertures **172** prevent the gas(es) from impinging directly on the substrate surface by slowing and re-directing the velocity profile of the flowing gases, as well as evenly distributing the flow of gas to provide an even distribution of gas across the surface of the substrate.

In one or more embodiments, the distribution plate **170** includes one or more embedded channels or passages **174** for housing a heater or heating fluid to provide temperature control of the lid assembly **140**. A resistive heating element (not shown) can be inserted within the passage **174** to heat the distribution plate **170**. A thermocouple can be connected to the distribution plate **170** to regulate the temperature thereof. The thermocouple can be used in a feedback loop to control electric current applied to the heating element, as described above.

Alternatively, a heat transfer medium can be passed through the passage **174**. The one or more passages **174** can contain a cooling medium, if needed, to better control temperature of the distribution plate **170** depending on the process requirements within the chamber body **112**. Any heat suitable transfer medium may be used, such as nitrogen, water, ethylene glycol, or mixtures thereof, for example.

In one or more embodiments, the lid assembly **140** can be heated using one or more heat lamps (not shown). Typically, the heat lamps are arranged about an upper surface of the distribution plate **170** to heat the components of the lid assembly **140** including the distribution plate **170** by radiation.

The blocker plate **175** may optionally be disposed between the second electrode **145** and the distribution plate **170** when present. The blocker plate **175** is removably mounted to a lower surface of the second electrode **145**. The blocker plate **175** may be in good thermal and electrical contact with the second electrode **145**. In one or more embodiments, the blocker plate **175** can be coupled to the second electrode **145** using a bolt or similar fastener. The blocker plate **175** can also be threaded or screwed onto an outer diameter of the second electrode **145**.

The blocker plate **175** includes a plurality of apertures **176** to provide a plurality of gas passages from the second

electrode **145** to the distribution plate **170**. The apertures **176** can be sized and positioned about the blocker plate **175** to provide a controlled and even flow distribution of gases to the distribution plate **170**.

The support assembly **180** can include a support member **185** to support a substrate (not shown in this view) for processing within the chamber body **112**. The support member **185** can be coupled to a lift mechanism **183** through a shaft **187** which extends through a centrally-located opening **114** formed in a bottom surface of the chamber body **112**. The lift mechanism **183** can be flexibly sealed to the chamber body **112** by a bellows **188** that prevents vacuum leakage from around the shaft **187**. The lift mechanism **183** allows the support member **185** to be moved vertically within the chamber body **112** between a process position and a lower transfer position. The transfer position is slightly below the slit valve opening **114** formed in a sidewall of the chamber body **112**.

In one or more embodiments, the support member **185** has a flat, circular surface or a substantially flat, circular surface for supporting a substrate to be processed thereon. The support member **185** may be constructed of aluminum. The support member **185** can include a removable top plate **190** made of some other material, such as silicon or ceramic material, for example, to reduce backside contamination of the substrate.

In one or more embodiments, the substrate (not shown) may be secured to the support member **185** using a vacuum chuck. In one or more embodiments, the substrate (not shown) may be secured to the support member **185** using an electrostatic chuck. An electrostatic chuck typically includes at least a dielectric material that surrounds an electrode **181**, which may be located in the support member **185** or formed as an integral part of the support member **185**. The dielectric portion of the chuck electrically insulates the chuck electrode from the substrate and from the remainder of the support assembly **180**.

In one embodiment, the electrode **181** that is coupled to a plurality of RF power bias sources **184**, **186**. The RF bias power sources **184**, **186** are coupled between the electrode **181** disposed in the support member **185**. The RF bias power excites and sustains a plasma discharge formed from the gases disposed in the processing region of the chamber body.

In the embodiment depicted in FIG. 1, the dual RF bias power sources **184**, **186** are coupled to the electrode **181** disposed in the support member **185** through a matching circuit **189**. The signal generated by the RF bias power **184**, **186** is delivered through matching circuit **189** to the support member **185** through a single feed to ionize the gas mixture provided in the plasma processing chamber **100**, thereby providing ion energy necessary for performing a deposition or other plasma enhanced process. The RF bias power sources **184**, **186** are generally capable of producing an RF signal having a frequency of from about 50 kHz to about 200 MHz and a power between about 0 Watts and about 5000 Watts. Additional bias power sources may be coupled to the electrode **181** to control the characteristics of the plasma as needed.

The support member **185** can include bores **192** formed therethrough to accommodate lift pins **193**, one of which is shown in FIG. 1. Each lift pin **193** is constructed of ceramic or ceramic-containing materials, and are used for substrate-handling and transport. The lift pin **193** is moveable within its respective bore **192** when engaging an annular lift ring **195** disposed within the chamber body **112**. The lift ring **195** is movable such that the upper surface of the lift-pin **193** can be extended above the substrate support surface of the

support member **185** when the lift ring **195** is in an upper position. Conversely, the upper surface of the lift-pins **193** is located below the substrate support surface of the support member **185** when the lift ring **195** is in a lower position. Thus, each lift-pin **193** is moved in its respective bore **192** in the support member **185** when the lift ring **195** moves from either the lower position to the upper position.

The support assembly **180** can further include an edge ring **196** disposed about the support member **185**. In one or more embodiments, the edge ring **196** is an annular member that is adapted to cover an outer perimeter of the support member **185** and protect the support member **185** from deposition. The edge ring **196** can be positioned on or adjacent the support member **185** to form an annular purge gas channel between the outer diameter of support member **185** and the inner diameter of the edge ring **196**. The annular purge gas channel can be in fluid communication with a purge gas conduit **197** formed through the support member **185** and the shaft **187**. The purge gas conduit **197** is in fluid communication with a purge gas supply (not shown) to provide a purge gas to the purge gas channel. Any suitable purge gas such as nitrogen, argon, or helium, may be used alone or in combination. In operation, the purge gas flows through the conduit **197**, into the purge gas channel, and about an edge of the substrate disposed on the support member **185**. Accordingly, the purge gas working in cooperation with the edge ring **196** prevents deposition at the edge and/or backside of the substrate.

The temperature of the support assembly **180** can be controlled by a fluid circulated through a fluid channel **198** embedded in the body of the support member **185**. In one or more embodiments, the fluid channel **198** is in fluid communication with a heat transfer conduit **199** disposed through the shaft **187** of the support assembly **180**. The fluid channel **198** is positioned about the support member **185** to provide a uniform heat transfer to the substrate receiving surface of the support member **185**. The fluid channel **198** and heat transfer conduit **199** can flow heat transfer fluids to either heat or cool the support member **185**. Any suitable heat transfer fluid may be used, such as water, nitrogen, ethylene glycol, or mixtures thereof. The support assembly **180** can further include an embedded thermocouple (not shown) for monitoring the temperature of the support surface of the support member **185**. For example, a signal from the thermocouple may be used in a feedback loop to control the temperature or flowrate of the fluid circulated through the fluid channel **198**.

The support member **185** can be moved vertically within the chamber body **112** so that a distance between support member **185** and the lid assembly **140** can be controlled. A sensor (not shown) can provide information concerning the position of support member **185** within chamber **100**.

In operation, the support member **185** can be elevated to a close proximity of the lid assembly **140** to control the temperature of the substrate being processed. As such, the substrate can be heated via radiation emitted from the distribution plate **170**. Alternatively, the substrate can be lifted off the support member **185** to close proximity of the heated lid assembly **140** using the lift pins **193** activated by the lift ring **195**.

A system controller (not shown) can be used to regulate the operations of the processing chamber **100**. The system controller can operate under the control of a computer program stored on a memory of a computer. The computer program may include instructions that enable the preclean process described below to be performed in the processing chamber **100**. For example, the computer program can

dictate the process sequencing and timing, mixture of gases, chamber pressures, RF power levels, susceptor positioning, slit valve opening and closing, wafer cooling and other parameters of a particular process.

FIG. 2 is a schematic top-view diagram of an illustrative multi-chamber processing system 200 that can be adapted to perform processes as disclosed herein having the processing chamber 100 coupled thereto. The system 200 can include one or more load lock chambers 202, 204 for transferring substrates into and out of the system 200. Typically, since the system 200 is under vacuum, the load lock chambers 202, 204 can “pump down” the substrates introduced into the system 200. A first robot 210 can transfer the substrates between the load lock chambers 202, 204, and a first set of one or more substrate processing chambers 212, 214, 216, 100 (four are shown). Each processing chamber 212, 214, 216, 100 is configured to perform at least one of substrate processing operations, such as an etching process, cyclical layer deposition (CLD), atomic layer deposition (ALD), chemical vapor deposition (CVD), physical vapor deposition (PVD), degas, orientation and other substrate processes. The position of the processing chamber 100 utilized to perform the etching process relative to the other chambers 212, 214, 216 is for illustration, and the position of the processing chamber 100 may be optionally be switched with any one of the processing chambers 212, 214, 216 if desired.

The first robot 210 can also transfer substrates to/from one or more transfer chambers 222, 224. The transfer chambers 222, 224 can be used to maintain ultra-high vacuum conditions while allowing substrates to be transferred within the system 200. A second robot 230 can transfer the substrates between the transfer chambers 222, 224 and a second set of one or more processing chambers 232, 234, 236, 238. Similar to processing chambers 212, 214, 216, 100, the processing chambers 232, 234, 236, 238 can be outfitted to perform a variety of substrate processing operations including the dry etch processes described herein any other suitable process including deposition, pre-clean, degas, and orientation, for example. Any of the substrate processing chambers 212, 214, 216, 100, 232, 234, 236, 238 can be removed from the system 200 if not necessary for a particular process to be performed by the system 200.

FIG. 3 illustrates a method 300 used to perform an etching process to etch a material layer disposed on a substrate with high selectivity and good profile control. The sequence described in FIG. 3 corresponds to the fabrication stages depicted in FIGS. 4A-4E, which is discussed below. FIGS. 4A-4E illustrate schematic cross-sectional views of a substrate 402 having a film stack 450 formed thereon during different stages of etching a material layer 406 disposed in the film stack 450 illustrated by the method 300.

The method 300 starts at block 302 by transferring a substrate, such as the substrate 402 depicted in FIG. 4A, into the processing chamber, such as the processing chamber 100 depicted in FIG. 1, or other suitable processing chamber. The substrate 402 may have a substantially planar surface, an uneven surface, or a substantially planar surface having a structure formed thereon. The substrate 402 shown in FIG. 4A includes a film stack 450 formed on the substrate 402. In one embodiment, the substrate 402 may be a material such as crystalline silicon (e.g., Si<100> or Si<111>), silicon oxide, strained silicon, silicon germanium, doped or undoped polysilicon, doped or undoped silicon wafers and patterned or non-patterned wafers silicon on insulator (SOI), carbon doped silicon oxides, silicon nitride, doped silicon, germanium, gallium arsenide, glass, sapphire. The substrate 402 may have various dimensions, such as 200 mm, 300 mm

or 450 mm diameter wafers, as well as, rectangular or square panels. Unless otherwise noted, embodiments and examples described herein are conducted on substrates with a 300 mm diameter or a 450 mm diameter.

In one embodiment, the film stack 450 includes a material layer 406 disposed on an etch stop layer 404 on the substrate 402. In one embodiment, the material layer 406 may be utilized to form a gate structure, shallow trench isolation (STI) structure, a contact structure or an interconnection structure in the front end or back end processes. In one embodiment, the method 300 may be performed on the material layer 406 to form a contact structure therein. In one embodiment, the etch stop layer 404 may be a silicon dielectric layer, such as a SiN layer. The material layer 406 may be a dielectric layer selected from a group consisting of an oxide layer, a nitride layer, titanium nitride layer, a composite of oxide and nitride layer, at least one or more oxide layers sandwiching a nitride layer, and combinations thereof, among others. Other suitable materials for the dielectric layer include undoped silicon glass (USG), such as silicon oxide or TEOS, boron-silicate glass (BSG), phosphorus-silicate glass (PSG), boron-phosphorus-silicate glass (BPSG) and combinations thereof. In an exemplary embodiment depicted herein, the material layer 406 is an undoped silicon glass (USG) layer. In one embodiment, the dielectric material layer 406 has a thickness between about 3000 Å to about 15000 Å, such as between about 4000 Å to about 12000 Å, for example about 10000 Å.

A patterned mask layer 411 is disposed on the material layer 406. The patterned mask layer 411 has an open feature 412 that exposes portions 413 of the material layer 406 for etching. In one embodiment, the mask layer 411 may be a hardmask layer, a photoresist mask or a combination thereof. The open feature 412 in the mask layer 411 is used as an etch mask to form open features 414 in the material layer 406 with desired aspect ratios. The open features 414 described herein may include trenches, vias, openings and the like. In one embodiment wherein the mask layer 411 is a hardmask layer, the mask layer 411 may be a material selected from a group consisting of silicon, silicon oxide, silicon nitride, silicon oxynitride, silicon carbide, amorphous carbon, and combinations thereof. In the embodiment wherein the mask layer 411 is a patterned photoresist layer, such as a lithographically patterned mask. The photoresist layer may be a positive tone photoresist, a negative tone photoresist, a UV lithography photoresist, an i-line photoresist, an e-beam resist (for example, a chemically amplified resist (CAR)) or other suitable photoresist. In an exemplary embodiment depicted in FIG. 4A, the patterned mask layer 411 is a combination of a photoresist layer 410 disposed on a hard mask layer 408 and the hard mask layer 408 is a silicon nitride layer.

At block 304, a main etching process is performed on the substrate 402 to etch the material layer 406 to a predetermined depth 418, leaving a desired thickness 422 of the material layer 406 on the substrate 402, to expose a portion of a surface 420 of the material layer 406, as shown in FIG. 4B. In one embodiment, the desired thickness 422 of the material layer 406 remaining on the substrate 402 may be between about 15 percent and about 25 percent of a total thickness 438 of the material layer 406.

During the main etching process, an etching gas mixture is supplied into the vacuum processing chamber 100 for processing. In one embodiment, the etching gas mixture includes at least one of a halogenated carbon gas or a halogenated hydrocarbon gas. Halogenated carbon gases may include, but are not limited to, fluorocarbons, hydro-

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fluorocarbons, fluorochlorocarbons, and hydrocarbons. The halogenated carbon gas and the halogenated hydrocarbon gas may have a formula $C_xH_yA_z$ (A representing halogen elements including at least one of F, Cl, Br, and the like) where x, y, and z are integers and x has a range between 1 and 10 and y has a range of between 0 and 22 and z has a range of between 0 and 22. Suitable examples of the halogenated carbon gas or the halogenated hydrocarbon gas include CH_2F_2 , CF_4 , CHF_3 , $CF_3(CF_2)_5(CH_2)_2SiCl_3$, CH_3F , C_4F_5 and the like. In an exemplary embodiment, the etching gas mixture includes a halogenated carbon gas. In one embodiment, the halogenated carbon gas used to etch the dielectric material layer **406** is C_4F_5 or C_4F_6 . The halogenated carbon gas or the halogenated hydrocarbon gas may be supplied into the vacuum processing chamber **100** at a volumetric flow rate between about 15 sccm and about 60 sccm.

In one embodiment, other types of gas, such as inert gas or carrier gas, may also be supplied in the etching gas mixture to assist carrying the etching gas mixture into the vacuum processing chamber **100**. Suitable examples of the inert gas or carrier gas include at least one of Ar, He, N_2 , O_2 , N_2O , NO_2 , NO, and the like. In one embodiment, the inert or carrier gas may be supplied into the vacuum processing chamber **100** at a volumetric flow rate between about 500 sccm and about 1000 sccm.

The halogenated carbon gas supplied in the main etching gas mixture may also serve as a polymer source to deposit a polymer layer on the substrate surface so as to protect an upper surface **415** of the mask layer **411** and sidewall **416** of the features being etched along the etching process. In the embodiments wherein the etching rate is not uniform across the substrate surface, the mask layer **411** having the composite film structure including the photoresist layer **410** and the hard mask layer **408** (e.g., a silicon nitride layer), may be consumed at different rates. During the main etching process with non-uniform etching rate, the photoresist layer **410** may already be consumed exposing the underlying hard mask layer **408** in some regions while still pertaining some photoresist layer **410** remaining on the substrate, thereby resulting the substrate surface having two types of the layers (e.g., the photoresist layer **410** in some regions while hard mask layer **408** in other regions of the substrate) exposed during etching. By using the halogenated carbon gas supplied in the etching gas mixture, the halogenated carbon gas may not only serve as an aggressive etchants to etch the material layer **406**, but also serve as a polymer source to form a polymer protection layer on the substrate for protection. As such, the selectivity between the material layer **406** and the hard mask layer **408** (e.g., the selectivity for a silicon oxide layer to a silicon nitride layer) may be improved and enhanced.

Several process parameters may also be regulated while the etching gas mixture is supplied into the vacuum processing chamber **100**. In one embodiment, the chamber pressure in the presence of the etching gas mixture is regulated. In one exemplary embodiment, a process pressure in the vacuum processing chamber **100** is regulated between about 5 mTorr to about 1000 mTorr, for example, between about 10 mTorr and about 200 mTorr. A first RF bias power at a frequency of about 60 MHz may be applied to maintain a plasma formed from the etching gas mixture. For example, a first RF bias power of between about 0 watts to about 500 watts may be applied to maintain a plasma inside the vacuum processing chamber **100**. A second RF bias power may also be applied to the processing chamber **100** of between about 200 watts to about 800 watts. The etching gas

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mixture may be flowed into the chamber at a rate between about 15 sccm to about 60 sccm. A substrate temperature is maintained between about 25 degrees Celsius to about 300 degrees Celsius, such as between about 50 degrees Celsius and about 110 degrees Celsius.

The main etching process performed at block **304** is configured to be a relatively fast etching process, e.g., an etching rate greater than 2000 Å per minute, as compared to the slow cyclic etching process, which will be described in detail below in block **308**. By utilizing this fast main etching process, the overall etching throughput may be maintained at a desired range, without significantly increase of manufacture cycle time contributed by the slow cyclic etching process subsequently performed.

At block **306**, a treatment process is performed to treat the exposed surface **420** along with an upper surface **415** of the mask layer **411** to alter the surface properties to facilitate removal of the material layer **406** in the subsequent chemical etching process. The treatment process performed at block **306** includes supplying a treatment gas mixture into the chamber **100**. A plasma is then formed from the treatment gas mixture to plasma treat the surfaces **420**, **415** of the substrate **402** to activate the material layer **406** into an excited state, forming a treated material layer **426** and a treated upper surface **428** of the mask layer **411**, as shown in FIG. **4C**, which may then easily react with chemical etching gases subsequently supplied into the processing chamber **100**, forming volatile gas byproducts which readily pumps out of the processing chamber **100**.

In one embodiment, the treatment gas mixture includes at least one of a hydrogen containing gas, a nitrogen containing gas, or an inert gas. It is believed that the hydrogen containing gas, the nitrogen containing gas, or inert gas supplied in the treatment gas mixture may assist increasing the lifetime of the ions in the plasma formed from the treatment gas mixture. Increased lifetime of the ions may assist reacting with and activating the material layer **426** on the substrate **402** more thoroughly, thereby enhancing the removal of the activated material layer **426** from the substrate **402** during the subsequent chemical etching process. In the embodiment wherein the hydrogen containing gas is utilized in the treatment gas mixture, the hydrogen atoms from the hydrogen containing gas may react with the silicon atoms contained in the material layer **406**, thereby forming weak and dangling bond of Si—H or Si—OH bond on the treated surface **420** of the treated material layer **426**. The treated material layer **426** with Si—H or Si—OH bond terminals may easily to be absorbed by other etchants subsequently supplied to the processing chamber **100**, thereby assisting ease of removal of the treated material layer **426** from the substrate surface.

In one embodiment, the hydrogen containing gas supplied into the processing chamber **100** includes at least one of H_2 , H_2O , and the like. The nitrogen containing gas supplied into the processing chamber **100** includes at N_2 , N_2O , NO_2 , NH_3 and the like. The inert gas supplied into the processing chamber **100** includes at least one of Ar, He, Kr, and the like. In an exemplary embodiment, the hydrogen containing gas supplied in the processing chamber **100** to perform the treatment process is H_2 gas, and the nitrogen containing gas supplied in the processing chamber **100** to perform the treatment process is N_2 gas and the inert gas is He or Ar.

During the plasma treatment process, several process parameters may be regulated to control the pretreatment process. In one exemplary embodiment, a process pressure in the processing chamber **100** is regulated between about 10 mTorr to about 5000 mTorr, such as between about 10 mTorr

and about 200 mTorr. A RF bias power at a frequency of about 13 MHz may be applied to maintain a plasma in the treatment gas mixture. For example, a RF bias power of about 20 Watts to about 200 Watts may be applied to maintain a plasma inside the processing chamber 100. The treatment gas mixture may be flowed into the chamber at a rate between about 200 sccm to about 800 sccm. A substrate temperature is maintained between about 25 degrees Celsius to about 300 degrees Celsius, such as between about 50 degrees Celsius and about 110 degrees Celsius.

In one embodiment, the substrate is subjected to the treatment process for between about 5 seconds to about 5 minutes, depending on the operating temperature, pressure and flow rate of the gas. For example, the substrate can be exposed to the pretreatment processes for about 30 seconds to about 90 seconds. In an exemplary embodiment, the substrate is exposed to the treatment process for about 90 seconds or less.

At block 306, a chemical etching process is performed to slowly remove the treated material layer 426 from the substrate 402, as shown in FIG. 4D. The chemical etching process is performed by supplying a chemical etching gas mixture through the plasma cavity 150 into the processing chamber 100 to form a remote plasma source in the plasma cavity 150 from the chemical etching gas mixture for etching the material layer 426. As the treated material layer 426 has been treated to have weak and dangling bonds with Si—H or Si—OH bond terminals on the surface, the aggressive etchants from the chemical etching gas mixture may easily attack the weak and dangling bonds with Si—H or Si—OH bond terminals and efficiently remove the treated material layer 426 from the substrate 402 during the chemical etching process.

In one embodiment, the chemical etching gas mixture used to remove the treated material layer 426 is a mixture of ammonia (NH₃) and nitrogen trifluoride (NF₃) gases. The amount of each gas introduced into the processing chamber may be varied and adjusted to accommodate, for example, the thickness of the material layer 416 to be removed, the geometry of the substrate being cleaned, the volume capacity of the plasma, the volume capacity of the chamber body, as well as the capabilities of the vacuum system coupled to the chamber body.

In one or more embodiments, the gases added to provide a chemical etching gas mixture having at least a 1:1 molar ratio of ammonia (NH₃) to nitrogen trifluoride (NF₃). In one or more embodiments, the molar ratio of the chemical etching gas mixture is at least about 3:1 (ammonia to nitrogen trifluoride). The gases are introduced in the chamber 100 at a molar ratio of from about 5:1 (ammonia to nitrogen trifluoride) to about 30:1. In yet another embodiment, the molar ratio of the chemical etching gas mixture is from about 5:1 (ammonia to nitrogen trifluoride) to about 10:1. The molar ratio of the chemical etching gas mixture can also fall between about 10:1 (ammonia to nitrogen trifluoride) and about 20:1. It is noted that the ration between the ammonia (NH₃) to nitrogen trifluoride (NF₃) may also be adjusted to improve the etching selectivity, including the selectivity between the material layer 426 and the hard mask layer 408 (e.g., selectivity of the silicon oxide layer to silicon nitride layer) and the selectivity between the material layer 426 and the underlying etch stop layer 404 (e.g., selectivity of the silicon oxide layer to silicon nitride layer).

As the treated material layer 426 remaining on the substrate has the relatively thin thickness 432 and the plasma is generated remotely in the plasma cavity 150, the etchants dissociated from the chemical etching gas mixture, the

remote source plasma as generated is relatively mild and gentle, so as to slowly, gently and gradually etch the remaining treated material layer 426 until the underlying substrate 402 is exposed. As compared to the fast etch rate of the main etching process performed at block 304, the chemical etching process performed at block 308 to remove the treated material layer 426 may be controlled to proceed at a slow rate, such as between about 1 Å per second and about 10 Å per second, so as to provide good control for the interface etching, thereby allowing precise etching end point for the treated material layer 426 being removed from the substrate 402. The chemical etching process is configured to chemically react and remove the treated material layer 426 from the substrate 402 without overly aggressive and physical bombarding, sputtering or biasing the substrate surface, thereby providing a mild etching process that slowly removes the treated material layer 426. Each cycle of the chemical etching process may only etch away about 25 Å of the treated material layer 426 so as to slowly remove the treated material layer 426 with good profile control, thereby reducing likelihood of over-etching to the substrate, profile deformation, or loss of CD control.

In the embodiment depicted in FIG. 4D, after the first cycle of the chemical etching, the treated material layer 426 may have a thickness 432 of about 900 Å left for further etching. In other words, in the first cycle of the chemical etching, the treated material layer 426 may have a thickness of between about 20 Å and about 50 Å removed from the substrate 402.

During the chemical etching process, several process parameters may be regulated to control the chemical etching process. In one exemplary embodiment, a process pressure in the processing chamber 100 is regulated to between about 10 mTorr and about 5000 mTorr, such as between about 800 mTorr and about 5 Torr. A RF source power is provided at a frequency of about 60 MHz to maintain a plasma in the chemical etching gas mixture. For example, a RF source power of about 20 Watts to about 50 Watts may be chemical etching gas mixture may be flowed into the chamber at a rate between about 15 sccm to about 60 sccm. A substrate temperature is maintained between about 25 degrees Celsius to about 300 degrees Celsius, such as between about 50 degrees Celsius and about 110 degrees Celsius. No bias power is provided during the chemical etching process to reduce ion bombardment.

At block 310, a transition process may be performed to remove etching residuals from the substrate surface. A transition gas mixture including a purge gas or carrier gas can be supplied to the processing chamber to assist pump/purge the etching residuals from the processing chamber. Suitable purge/carrier gases include argon, helium, hydrogen, nitrogen, or mixtures thereof. The operating pressure within the chamber can be variable. Application of the RF source or RF bias powers may be eliminated at this pump/purge process, e.g., without applying a RF power during the transition process. The purge/carrier gas can be used to facilitate the pump/purge of the etching residuals from the processing chamber. In one embodiment, the substrate is subjected to perform transition process for between about 5 seconds to about 5 minutes. For example, the substrate can be exposed for about 5 seconds to about 10 seconds. In an exemplary embodiment, the substrate is exposed for about 5 seconds.

It is noted that blocks 306, 308 and 310 may be repeatedly (i.e., cyclically) performed, as indicated by the loop 312 in FIG. 3, until a desired target depth 438 of the feature 414 is formed in the material layer 406 exposing a surface 436 of

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the underlying etch stop layer **404** on the substrate **402**, as shown in FIG. **4E**. In one embodiment, the desired target depth **438** may be between about 40 nm and about 60 nm. The repeated processes may cyclically and incrementally etch the material layer **406** without over aggressively attacking the material layer **406**, thereby providing a good interface etching control and a proper etch stop endpoint. Incremental etching with repetitive treatment, chemical etching process and the transition process that improves feature verticality and promotes etching selectivity between the material layer **406** and the etch stop layer **404**, thereby enhancing accuracy of mask to feature CD transfer between the mask **411** and the material layer **406**. In one embodiment, the feature **411** may have a critical dimension less than 40 nm and an aspect ratio greater than 7, such as between about 5 and about 10.

Thus, a method and an apparatus for cyclically etching a material layer with high selectivity and good profile control are provided. The method may allow a proper etching stop endpoint with good interface control while providing an acceptable range of overall etching throughput. The etching process utilizing a main etching process along with a cyclic three-step etching process, e.g., a treatment process, a chemical cleaning process and a transition process, to etch features into a material layer. The main etching process along with the cyclic three step etching process can efficiently form features in a material layer with high selectivity and good profile control without significantly degrading of the overall process time.

While the foregoing is directed to embodiments of the present invention, other and further embodiments of the invention can be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

The invention claimed is:

1. A method for etching a feature in a material layer comprising:

- (a) performing a main etching process in a processing chamber by supplying a main etching gas mixture and applying a RF bias power to etch an oxide layer exposed by a patterned mask layer disposed on a substrate, forming a feature with a first predetermined depth in the oxide layer;
- (b) subsequently performing a treatment process on the substrate by supplying a treatment gas mixture into the processing chamber to treat the etched feature in the oxide layer;
- (c) subsequently performing a chemical etching process on the substrate by supplying a chemical etching gas mixture different from the main etching gas mixture without applying the RF bias power into the processing chamber to continue etching the feature in the oxide layer, wherein the chemical etching gas includes at least an ammonium gas and a nitrogen trifluoride, wherein the chemical etching process further etches the feature to a second predetermined depth;
- (d) subsequently performing a transition process on the etched substrate by supplying a transition gas mixture into the processing chamber; and
- (e) repeatedly performing step (b)-step (d) to etch the feature in the oxide layer until an underlying substrate is exposed.

2. The method of claim 1, wherein the treatment gas mixture includes at least a hydrogen containing gas, a nitrogen containing gas or an inert gas.

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3. The method of claim 1, wherein the transition gas mixture includes at least a hydrogen containing gas, a nitrogen containing gas or an inert gas.

4. The method of claim 1, wherein the first predetermined depth is about 15 percent to about 25 percent to a total thickness of the oxide layer prior to etching.

5. The method of claim 1, wherein a ratio of the first predetermined depth to the second predetermined depth is between about 2:1 to about 8:1.

6. The method of claim 1, wherein performing the main etching process further comprises:

supplying the main etching gas mixture including C_4F_8 or C_4F_6 ; and

applying a RF bias power to the etching gas mixture to generate a plasma.

7. The method of claim 1, wherein performing the treatment process on the substrate further comprises:

applying a RF bias power to the treatment gas mixture.

8. The method of claim 1, wherein performing the chemical etching process on the substrate further comprises:

applying a RF source power to the chemical etching gas mixture.

9. The method of claim 1, wherein performing the transition process further comprises:

supplying the transition gas mixture to remove etching residual without applying a RF power.

10. The method of claim 1, wherein performing the chemical etching process on the substrate further comprises:

supplying the ammonium gas and the nitrogen trifluoride in the chemical etching gas mixture a molar ratio of from about 5:1 to about 30:1.

11. The method of claim 1, further comprising:

maintaining a substrate temperature between about 50 degrees and about 110 degrees Celsius.

12. A method for etching a feature in a material layer comprising:

(a) transferring a substrate having a material layer disposed thereon in a processing chamber, wherein the material layer has a patterned mask layer disposed thereon exposing a portion of the material layer;

(b) supplying a main etching gas mixture and applying a RF bias power into the processing chamber to etch the portion of the material layer exposed by the patterned mask layer to form a feature in the material layer until to a first predetermined depth of the feature is formed in the material layer, wherein the etching gas mixture includes a halogenated carbon gas;

(c) supplying a treatment gas mixture to treat the feature formed in the material layer, wherein the treatment gas mixture includes an inert gas; and

(d) supplying a chemical etching gas mixture different from the etching gas mixture without applying the RF bias power into the processing chamber to continue etching the feature formed in the material layer until the substrate underneath the material layer is exposed, wherein the chemical etching gas mixture includes at least an ammonium gas and a nitrogen trifluoride; and

(e) supplying a transition gas mixture to the processing chamber, wherein the transition gas mixture includes an inert gas.

13. The method of claim 12, wherein the first predetermined depth of the feature etched away from the material layer while supplying the etching gas mixture is between about 15 percent and about 25 percent from the total thickness of the material layer.

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14. The method of claim 12, wherein the ammonium gas and the nitrogen trifluoride in the chemical etching gas mixture a molar ratio of from about 5:1 to about 30:1.

15. The method of claim 12, further comprising:
repeatedly performing steps (c) to (e) until a second
predetermined depth of the feature is formed in the
material layer. 5

16. The method of claim 12, further comprises:
applying a RF bias power to the treatment gas mixture;
applying a RF source power to the chemical etching gas
mixture; and 10
supplying the transition gas mixture to remove etching
residual without applying a RF power.

17. The method of claim 12, wherein the material layer is
a oxide layer configured to form a contact structure. 15

18. A method for etching a feature in a material layer
comprising:

(a) transferring a substrate having a material layer dis-
posed thereon into a processing chamber, wherein the
material layer has a patterned mask layer disposed 20
thereon exposing a portion of the material layer;

(b) supplying a C_4F_8 or C_4F_6 gas and applying a first RF
bias power into the processing chamber to etch the

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portion of the material layer exposed by the patterned
mask layer to form a feature in the material layer until
to a first predetermined depth of the feature is formed
in the material layer, wherein the first predetermined
depth of the feature is between about 15 percent and
about 25 percent to a total thickness of the material
layer;

(c) subsequently supplying a Ar or He gas while applying
a second RF bias power to treat the feature formed in
the material layer;

(d) subsequently supplying a chemical etching gas mix-
ture including at least an ammonium gas and a nitrogen
trifluoride while applying a first RF source power
without the second RF bias power to etch the treated
feature to continue etching the treated feature formed in
the material layer until the substrate underneath the
material layer is exposed; and

(e) subsequently supplying a Ar or He gas to the process-
ing chamber without applying a RF power.

19. The method of claim 18, further comprising:
repeatedly performing steps (c) to (e) until a desired depth
of the feature is formed in the material layer.

* * * * *

UNITED STATES PATENT AND TRADEMARK OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 9,543,163 B2
APPLICATION NO. : 14/059416
DATED : January 10, 2017
INVENTOR(S) : Mang-Mang Ling et al.

Page 1 of 1

It is certified that error appears in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

In the Specification

In Column 2, Line 67, delete “perfect” and insert -- percent --, therefor.

In Column 11, Line 10, delete “C₄F₅” and insert -- C₄F₈ --, therefor.

In Column 11, Line 13, delete “C₄F₅” and insert -- C₄F₈ --, therefor.

In the Claims

In Column 18, Line 6, in Claim 18, delete “perfect” and insert -- percent --, therefor.

Signed and Sealed this
Fourteenth Day of March, 2017



Michelle K. Lee
Director of the United States Patent and Trademark Office